



31013

PATENT  
33168-2130

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : John Skoufis  
Serial No. : 09/879,613  
Filed : 06/12/2001  
For : PEROXIDE PRESERVATION  
Group Art Unit : 3728  
Examiner : Mohandesi, Jila M.

1177 Avenue Of The Americas  
New York, NY 10036

DECLARATION UNDER 37 C.F.R. §1.132

**MAIL STOP AMENDMENT**

Commissioner For Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, John Skoufis, the inventor named in the above-identified U.S. patent application, hereby declare and state as follows:

1. ITW Texwipe Inc. is the Assignee of the above-identified patent application. I was the Director of Research and Development of The Texwipe Company, the predecessor of ITW Texwipe as the owner of this patent application, in the years 2000 and 2001, when the invention described in the above-identified patent application was made.

2. During 2000, Texwipe was packaging and selling brushes made of polyvinyl acetal (PVA) in the semiconductor manufacturing industry, where the brushes were used for scrubbing semiconductor wafers. The preservation method used to prevent bacterial growth and contamination of the PVA brushes was electron-beam radiation.

3. At that time, the methods known for preserving new wet PVA brushes included the use of electron-beam ("e-beam") radiation, gamma radiation, and the use of a small quantity of ammonium hydroxide. Each of those methods had drawbacks, and we wanted to use a different method without those drawbacks. We decided to test the possible use of hydrogen peroxide.

4. Our prior experience and the general knowledge in the semiconductor manufacturing industry was that hydrogen peroxide could be used as a bactericide at a concentration of 1% to 3%, with the higher concentrations being preferred in order to assure effectiveness.

5. In order to determine the proper concentration for use with PVA sponges, we performed a series of tests, both internally, and through outside laboratories.

6. The first test was performed by Balazs Laboratory in Sunnyvale, California in November, 2000. In that test, a solution of one percent (1%) electronic grade hydrogen peroxide

in de-ionized water was used as a preservative in the packages with the PVA brush sponges. The test samples were prepared at Texwipe by washing them very thoroughly and removing the wash water by centrifuge. Then the samples were given a final rinse with pure de-ionized water containing 1% hydrogen peroxide, double-bagged in plastic bags, and shipped to the Belazs Laboratories, where the bags were held for around 24 hours, opened and tested by leaching the brushes and measuring the metallic ion concentrations. All procedures were conducted under clean room conditions.

7. The results of this first test are shown in Exhibit 1, which is a copy of the test results together with an e-mail summarizing and reporting the results of the test to Texwipe.

8. In general, customers in the industry at the time of the tests required the concentration of metal ions in PVA brushes to be below ten parts per million and preferably less than one part per million. As it can be seen in the test results of Exhibit 1, several of the metal ion counts were much higher than those levels, and were unacceptable.

9. The PVA brushes tested had been cleaned very vigorously and thoroughly. Nevertheless, we found that they apparently contained a much higher level of metallic particles

than we had anticipated, and that the one percent (1%) hydrogen peroxide solution was creating the unacceptably high levels of metallic ions.

10. We decided to test a hydrogen peroxide concentration one order of magnitude lower, namely, 0.1%. Because this was much lower than prior recommended levels, we did not expect that concentration to be effective in killing bacteria.

11. The test samples were prepared in the same way as the samples for the tests reported in Exhibit 1, except for the concentration of hydrogen peroxide used, and except for a preliminary wash also using hydrogen peroxide. Later testing indicated that the preliminary hydrogen peroxide wash was not necessary, and it was not used in the final process.

12. Exhibit 2 is a copy of the test results using a 0.1% concentration of hydrogen peroxide in de-ionized water. The test was a standard test aimed at determining sterilization effectiveness. The test, which was performed in January of 2001, indicate that a brush stored in de-ionized water without peroxide developed twenty-three Colony Forming Units (CFU - standard units for such a test), and packages with 0.1% peroxide formed 0 CFU's. In general, a formation of from 0 to 3 CFU's is considered acceptable. Therefore, the brush without the

peroxide produced greatly excessive numbers of bacterial colonies, but the sterilization of those that were treated with 0.1% peroxide was completely adequate.

13. We also did not know whether the metallic ion formation levels would be sufficiently low using a 0.1% solution. Exhibit 3 is a test report from Belazs of metal ions developed by the 0.1% peroxide solution used to package PVA brushes, along with the corresponding measurements for other concentrations. Certain irrelevant pages of all of the test results have been omitted for the sake of clarity. The test samples were prepared in the manner described above for the first test.

14. The test results on pages 2 and 3 of Exhibit 3 are so-called "blank" test results produced by the test equipment when testing pure water. These measurements were taken to verify the accuracy of the equipment. The test results on pages 4 and 5 are the results of "blind" tests on three different samples whose makeup was not known to the testing personnel. The first lot, whose results are listed in the first column on the left, used a solution of 1% peroxide; the middle column showed the results of testing a sample using 0.5% peroxide, and the right-hand column showed the results of testing a sample using 0.1% peroxide. It can be seen that the

metal ion counts in the first column are totally unacceptable but those in the right-hand column are fully acceptable. Therefore, the metal ion counts produced by the 0.1% solution were well within an acceptable range.

15. We also found, by separate tests, that the hydrogen peroxide in the packages decomposed substantially completely within a time period of thirty minutes to one hour after packaging the brush. This was totally surprising to us. It also was totally surprising to us that, despite the quick decomposition of the hydrogen peroxide, it still was sufficiently effective to sterilize the brush. Written reports of these test results have been destroyed and no longer are available.

16. The surprisingly rapid decomposition of the hydrogen peroxide after the package was sealed gave us an unexpected advantage for the product.

17. When a customer uses a PVA brush in scrubbing semiconductor wafers, it first must compensate for any extraneous materials in the brush. Therefore, when any preservative, such as ammonium hydroxide or hydrogen peroxide, remains in the brush when the customer receives it, it must be compensated for before the brush can be used.

18. With certain preservation methods, such as the e-beam and gamma radiation methods described above, there are no preservatives which remain in the package with the brush delivered to the customer. This is a distinct advantage in that the customer need not compensate for any preservative. However, those radiation preservation methods are relatively expensive and difficult to use effectively.

19. Therefore, it turned out to be a great advantage to customers receiving brushes using the invention that the brushes were fully sterilized, but had no residual preservative, and did not have the uncertainty of effectiveness sometimes accompanying radiation type sterilization procedures.

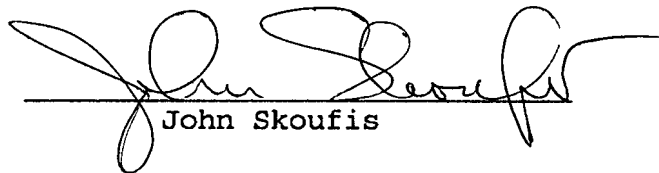
20. Because the peroxide content deteriorated within a maximum of one hour, we were able to guarantee that a customer would receive the brush without any preservative remaining in the package, even if the brush were shipped overnight right after it had been packaged.

21. This was a benefit that we did not expect to achieve when we reduced the concentration of hydrogen peroxide to the 0.1% levels.

22. Further testing was done after the test reported in Exhibit 3 to make certain that the advantages obtained

experimentally were also obtained in practice when the method was used in production, and we found that they were.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



John Skoufis

October 2, 2007

#### Attachments

- Exhibit 1 - Test Results - metal ions using 1% peroxide
- Exhibit 2 - Test Results - bacterial activity using 0.1% peroxide
- Exhibit 3 - Test Results - metal ions - blind test of 1%, 0.5% and 0.1% peroxide solutions

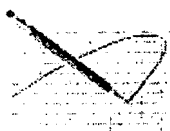


**EXHIBIT 1**

**TEST RESULTS**

**METAL IONS USING**

**1% PEROXIDE**



Tom J. Pavlik/Texwipe  
11/10/2000 06:06 PM

Steven J. Paley/Texwipe@Texwipe, John  
To Skoufis/Texwipe@Texwipe, Himansu  
Bhattacharjee/Texwipe@Texwipe, Duane

cc

bcc

Subject Summary of Balazs Data on PVA Brushes

Take a look at the attached spreadsheet. I provided the raw data as well as its equivalent when calculated with the dry weight of the brush. Also, I included the Ebara spec for the various metals of concern to them:



ICP-MS Testing.xls

All testing done by Balazs Labs using their standard procedures



Date 11/1/2000  
PVA Type TX6HP  
Laundering USR  
Dry Wt.(grams) 17.8 Calculated wt.  
Conditions 0.35%citric+ 38mm OD, 18mm ID, 208 L  
1%peroxide

**Trace Anions**

(ppm)	by dry wt.
Aluminum	360
Barium	4
Cadmium	10
Calcium	1854
Chromium	79
Cobalt	1
Copper	3314
Iron	1123
Lead	6
Magnesium	3707
Manganese	38
Nickel	219
Silicon	2809
Sodium	129
Strontium	12
Tin	3
Titanium	7
Tungsten	7
Zinc	6179

**ppm  
raw data**

Aluminum	6.4
Barium	0.07
Cadmium	0.17
Calcium	33
Chromium	1.4
Cobalt	0.02
Copper	59
Iron	20
Lead	0.11
Magnesium	66
Manganese	0.68
Nickel	3.9
Silicon	50
Sodium	2.3
Strontium	0.21
Tin	0.05
Titanium	0.13
Tungsten	0.13
Zinc	110

Two CCS processed  
brushes tested by Ebara on  
8/2000

Aluminum	4.02	16.25
Calcium	51.5	57
Chromium	1.25	0.29
Copper	0.21	78.5
Iron	7.65	5.04
Lead	0.16	0.6
Magnesium	73	68.5
Manganese	0.19	0.6
Nickel	0.15	0.15
Potassium	5.49	1.5
Sodium	4.29	1.1

**EXHIBIT 2**

**TEST RESULTS**

**BACTERIAL ACTIVITY USING**

**0.1% PEROXIDE**

John Skoufis/Texwipe

02/06/2001 09:31 AM

To Tom J. Pavlik/Texwipe@Texwipe

Mario Jakus/Texwipe@Texwipe, Steven J.

.cc Paley/Texwipe@Texwipe, Richard A.

Crimmins/Texwipe@Texwipe, Duane

bcc

Subject Sterility Testing of Peroxide experiments

The following results were obtained for Lot 01-01-58060-6016:

A. Without Peroxide: 23 CFU

B. With Peroxide: 0 CFU

The addition of a small amount of peroxide appears to help in this experiment that ran a preliminary peroxide wash and a 0.1% peroxide last rinse.

This is not to be taken as conclusive since we have seen other instances of contamination. In addition, the 23 CFU count is high in relation to what we have seen with other untreated samples. We would normally expect that the preliminary peroxide wash would kill any bacteria and the following washes would be with bacteria free water. At the very least, we would not expect the CFU count to be greater than 1.

Additional runs would be needed to determine the efficacy of the treatment and process. Metals testing is due for completion this week and will be reported when received.

**EXHIBIT 3**

**TEST RESULTS**

**METAL IONS BLIND TEST**

**OF 1%, 0.5% AND 0.1%**

**PEROXIDE SOLUTIONS**



Parameter	DL	Units	Sample Identification / Site
<b>68 Trace Elements - Blank</b>			
			<u>UPW BLANK</u>
Aluminum (Al)	0.05	ppb (ug/L)	*
Antimony (Sb)	0.02	ppb (ug/L)	*
Arsenic (As)	0.2	ppb (ug/L)	*
Barium (Ba)	0.01	ppb (ug/L)	*
Beryllium (Be)	0.04	ppb (ug/L)	*
Bismuth (Bi)	0.04	ppb (ug/L)	*
Boron (B)	0.5	ppb (ug/L)	*
Cadmium (Cd)	0.03	ppb (ug/L)	*
Calcium (Ca)	3	ppb (ug/L)	*
Cerium (Ce)	0.01	ppb (ug/L)	*
Cesium (Cs)	0.02	ppb (ug/L)	*
Chromium (Cr)	0.03	ppb (ug/L)	*
Cobalt (Co)	0.02	ppb (ug/L)	*
Copper (Cu)	0.05	ppb (ug/L)	*
Dysprosium (Dy)	0.04	ppb (ug/L)	*
Erbium (Er)	0.02	ppb (ug/L)	*
Europium (Eu)	0.02	ppb (ug/L)	*
Gadolinium (Gd)	0.04	ppb (ug/L)	*
Gallium (Ga)	0.04	ppb (ug/L)	*
Germanium (Ge)	0.05	ppb (ug/L)	*
Gold (Au)	0.05	ppb (ug/L)	*
Hafnium (Hf)	0.03	ppb (ug/L)	*
Holmium (Ho)	0.01	ppb (ug/L)	*
Indium (In)	0.02	ppb (ug/L)	*
Iridium (Ir)	0.06	ppb (ug/L)	*
Iron (Fe)	5	ppb (ug/L)	*
Lanthanum (La)	0.01	ppb (ug/L)	*
Lead (Pb)	0.05	ppb (ug/L)	*
Lithium (Li)	0.03	ppb (ug/L)	*
Lutetium (Lu)	0.01	ppb (ug/L)	*
Magnesium (Mg)	0.02	ppb (ug/L)	*
Manganese (Mn)	0.03	ppb (ug/L)	*
Mercury (Hg)	0.05	ppb (ug/L)	*
Molybdenum (Mo)	0.05	ppb (ug/L)	*
Neodymium (Nd)	0.02	ppb (ug/L)	*
Nickel (Ni)	0.05	ppb (ug/L)	*
Niobium (Nb)	0.02	ppb (ug/L)	*

This report, including any attachments, has been reviewed and approved by:

Stephen O'Neil

Laboratory Director

These results were obtained by following standard laboratory procedures and are only representative of the samples as received by the laboratory. The liability of BALAZS ANALYTICAL LABORATORY shall not exceed the amount paid for this report. In no event shall BALAZS ANALYTICAL LABORATORY be liable for special or consequential damages. Client agrees not to use Balazs' name in reporting results obtained from tests performed by Balazs without first obtaining Balazs written consent as to such use. Report shall not be reproduced except in full, without the written approval of BALAZS ANALYTICAL LABORATORY.

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Parameter	DL	Units	Sample Identification / Site
<b>68 Trace Elements - Blank</b>			
			<u>UPW BLANK</u>
Osmium (Os)	0.02	ppb (ug/L)	*
Palladium (Pd)	0.06	ppb (ug/L)	*
Platinum (Pt)	0.5	ppb (ug/L)	*
Potassium (K)	5	ppb (ug/L)	*
Praseodymium (Pr)	0.01	ppb (ug/L)	*
Rhenium (Re)	0.06	ppb (ug/L)	*
Rhodium (Rh)	0.02	ppb (ug/L)	*
Rubidium (Rb)	0.01	ppb (ug/L)	*
Ruthenium (Ru)	0.05	ppb (ug/L)	*
Samarium (Sm)	0.04	ppb (ug/L)	*
Scandium (Sc)	0.2	ppb (ug/L)	*
Selenium (Se)	7	ppb (ug/L)	*
Silicon (Si)	0.5	ppb (ug/L)	*
Silver (Ag)	0.03	ppb (ug/L)	*
Sodium (Na)	0.06	ppb (ug/L)	*
Strontium (Sr)	0.01	ppb (ug/L)	*
Tantalum (Ta)	0.02	ppb (ug/L)	*
Tellurium (Te)	0.04	ppb (ug/L)	*
Terbium (Tb)	0.02	ppb (ug/L)	*
Thallium (Tl)	0.05	ppb (ug/L)	*
Thorium (Th)	0.02	ppb (ug/L)	*
Thulium (Tm)	0.01	ppb (ug/L)	*
Tin (Sn)	0.02	ppb (ug/L)	*
Titanium (Ti)	0.1	ppb (ug/L)	*
Tungsten (W)	0.2	ppb (ug/L)	*
Uranium (U)	0.02	ppb (ug/L)	*
Vanadium (V)	0.03	ppb (ug/L)	*
Ytterbium (Yb)	0.03	ppb (ug/L)	*
Yttrium (Y)	0.02	ppb (ug/L)	*
Zinc (Zn)	0.06	ppb (ug/L)	*
Zirconium (Zr)	0.05	ppb (ug/L)	*

Silicon analyzed using Colorimetry Process. All other elements are analyzed by ICP-MS.

This report, including any attachments, has been reviewed and approved by:

Stephen O'Neil

Laboratory Director

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Fax : (201) 327-6945

Report Date: 08-Jan-01  
Order Date: 22-Dec-00  
Work Order: 00-07263-03  
Client P.O.: 62063  
Release:  
Page: 4 of 6

Parameter	DL	Units	Sample Identification / Site		
Leachable 68 Trace Elements			LOT #L150AK-001	LOT #L150AK-002	LOT #L150AK-003
<b>Leachable TM by ICP-MS</b>					
Aluminum (Al)	0.05	ppb (ug/L)	3.2	1.5	0.32
Antimony (Sb)	0.02	ppb (ug/L)	*	*	*
Arsenic (As)	0.2	ppb (ug/L)	*	*	*
Barium (Ba)	0.01	ppb (ug/L)	*	*	*
Beryllium (Be)	0.04	ppb (ug/L)	*	*	*
Bismuth (Bi)	0.04	ppb (ug/L)	*	*	*
Boron (B)	0.5	ppb (ug/L)	*	*	*
Cadmium (Cd)	0.03	ppb (ug/L)	*	*	*
Calcium (Ca)	3	ppb (ug/L)	120	58	*
Cerium (Ce)	0.01	ppb (ug/L)	*	*	*
Cesium (Cs)	0.02	ppb (ug/L)	*	*	*
Chromium (Cr)	0.03	ppb (ug/L)	0.12	*	*
Cobalt (Co)	0.02	ppb (ug/L)	*	*	*
Copper (Cu)	0.05	ppb (ug/L)	18	4.3	0.67
Dysprosium (Dy)	0.04	ppb (ug/L)	*	*	*
Erbium (Er)	0.02	ppb (ug/L)	*	*	*
Europium (Eu)	0.02	ppb (ug/L)	*	*	*
Gadolinium (Gd)	0.04	ppb (ug/L)	*	*	*
Gallium (Ga)	0.04	ppb (ug/L)	*	*	*
Germanium (Ge)	0.05	ppb (ug/L)	*	*	*
Gold (Au)	0.05	ppb (ug/L)	*	*	*
Hafnium (Hf)	0.03	ppb (ug/L)	*	*	*
Holmium (Ho)	0.01	ppb (ug/L)	*	*	*
Indium (In)	0.02	ppb (ug/L)	*	*	*
Iridium (Ir)	0.06	ppb (ug/L)	*	*	*
Iron (Fe)	5	ppb (ug/L)	77	28	*
Lanthanum (La)	0.01	ppb (ug/L)	*	*	*
Lead (Pb)	0.05	ppb (ug/L)	*	*	*
Lithium (Li)	0.03	ppb (ug/L)	*	*	*
Lutetium (Lu)	0.01	ppb (ug/L)	*	*	*
Magnesium (Mg)	0.02	ppb (ug/L)	34	26	0.32
Manganese (Mn)	0.03	ppb (ug/L)	0.66	0.24	*
Mercury (Hg)	0.05	ppb (ug/L)	*	*	*
Molybdenum (Mo)	0.05	ppb (ug/L)	*	*	*
Neodymium (Nd)	0.02	ppb (ug/L)	*	*	*

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Stephen O'Neil

Laboratory Director

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Report Date: 08-Jan-01  
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Client P.O.: 62063  
Release:  
Page: 5 of 6

<u>Parameter</u>	<u>DL</u>	<u>Units</u>	<u>Sample Identification / Site</u>		
<i>Leachable 68 Trace Elements</i>					
			<u>LOT #L150AK-001</u>	<u>LOT #L150AK-002</u>	<u>LOT #L150AK-003</u>
<i>Leachable TM by ICP-MS</i>					
Nickel (Ni)	0.05	ppb (ug/L)	*	*	*
Niobium (Nb)	0.02	ppb (ug/L)	*	*	*
Osmium (Os)	0.02	ppb (ug/L)	*	*	*
Palladium (Pd)	0.06	ppb (ug/L)	*	*	*
Platinum (Pt)	0.5	ppb (ug/L)	*	*	*
Potassium (K)	5	ppb (ug/L)	*	*	*
Praseodymium (Pr)	0.01	ppb (ug/L)	*	*	*
Rhenium (Re)	0.06	ppb (ug/L)	*	*	*
Rhodium (Rh)	0.02	ppb (ug/L)	*	*	*
Rubidium (Rb)	0.01	ppb (ug/L)	*	*	*
Ruthenium (Ru)	0.05	ppb (ug/L)	*	*	*
Samarium (Sm)	0.04	ppb (ug/L)	*	*	*
Scandium (Sc)	0.2	ppb (ug/L)	*	*	*
Selenium (Se)	7	ppb (ug/L)	*	*	*
Silicon (Si)	0.5	ppb (ug/L)	36	21	4.4
Silver (Ag)	0.03	ppb (ug/L)	*	*	*
Sodium (Na)	0.06	ppb (ug/L)	1.4	0.86	1.2
Strontium (Sr)	0.01	ppb (ug/L)	0.04	*	*
Tantalum (Ta)	0.02	ppb (ug/L)	*	*	*
Tellurium (Te)	0.04	ppb (ug/L)	*	*	*
Terbium (Tb)	0.02	ppb (ug/L)	*	*	*
Thallium (Tl)	0.05	ppb (ug/L)	*	*	*
Thorium (Th)	0.02	ppb (ug/L)	*	*	*
Thulium (Tm)	0.01	ppb (ug/L)	*	*	*
Tin (Sn)	0.02	ppb (ug/L)	*	*	*
Titanium (Ti)	0.1	ppb (ug/L)	*	*	*
Tungsten (W)	0.2	ppb (ug/L)	*	*	*
Uranium (U)	0.02	ppb (ug/L)	*	*	*
Vanadium (V)	0.03	ppb (ug/L)	*	*	*
Ytterbium (Yb)	0.03	ppb (ug/L)	*	*	*
Yttrium (Y)	0.02	ppb (ug/L)	*	*	*
Zinc (Zn)	0.06	ppb (ug/L)	22	8.2	0.29
Zirconium (Zr)	0.05	ppb (ug/L)	*	*	*

## Conditions

Pre-Cleaning Solution

None

None

None

This report, including any attachments, has been reviewed and approved by:

Stephen O'Neil

Laboratory Director

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Page: 6 of 6

<u>Parameter</u>	<u>DL</u>	<u>Units</u>	<u>Sample Identification / Site</u>		
<b>Leachable 68 Trace Elements</b>					
			<u>LOT #L150AK-001</u>	<u>LOT #L150AK-002</u>	<u>LOT #L150AK-003</u>
<b>Conditions</b>					
Leaching Volume, ml			1,000	1,000	1,000
Leaching Temperature, C			Ambient	Ambient	Ambient
Sample Weight, grams			82.794	89.885	85.618
Surface Area, cm2			--	--	--
Leaching Time, hrs			24	24	24

Silicon analyzed using Colorimetry Process. All other elements are analyzed by ICP-MS.

- \*\* Brush was received wet. The wet weight of sample was recorded before leaching.
- The 12.5 cm long PVA brushes were leached in pre-cleaned polypropylene containers.
  - A leach blank was also prepared under identical conditions.
  - The result values are blank subtracted.
  - The leach conditions are given in the report.

\* = Analysis revealed that the analyte was not found at or above the detection limit DL = Detection Limit.

**Report Notes:** This is the final report. Silicon results have been added.

This report, including any attachments, has been reviewed and approved by:

Stephen O'Neil

Laboratory Director

These results were obtained by following standard laboratory procedures and are only representative of the samples as received by the laboratory. The liability of BALAZS ANALYTICAL LABORATORY shall not exceed the amount paid for this report. In no event shall BALAZS ANALYTICAL LABORATORY be liable for special or consequential damages. Client agrees not to use Balazs' name in reporting results obtained from tests performed by Balazs without first obtaining Balazs written consent as to such use. Report shall not be reproduced except in full, without the written approval of BALAZS ANALYTICAL LABORATORY.

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